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LLNL-TR-416360

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August 31, 2009

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This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

Formulation and Characterization of LX-17-2 from new FK 800 binder and WA, ATK, and BAE TATBs

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Background:

Currently LLNL has no Kel-F 800 or wet-aminated TATB reserves for formulation. Although both materials are soon to be commercially available, their synthesis processes have changed and the explosive must be re-evaluated.

In 2000 3M phased out the uses of perfluorooctanoyl (C8) derivatives due to environmental persistence and bioaccumulation issues. A C8 derivative was used as an emulsifier for making Kel F-800. In 2001 Kel F-800 was scheduled to be discontinued and the last Kel F-800 run was made in early 2002. LANL ordered 2M\$ worth of Kel-F 800 for reserves and Pantex purchased several hundred pounds to satisfy mock needs. After four years, 3M has decided to introduce a Kel-F 800-like polymer based on a new emulsifier using the same chlorotrifluoroethylene and vinylidene fluoride monomers and emulsion polymerization process. They have produced 3 batches and claim the "new" FK-800 is indistinguishable from the "old" Kel-F 800 in any of their testing parameters. In June-July 2006 3M scaled up a batch of about 800 pounds and have test quantities available. We have samples of the new FK-800 for evaluation. [1,2]

Neither wet nor dry-aminated TATB has been synthesized in the US in any significant quantity since about 1985 and significant quantities of LX-17-1 has not been formulated since about 1990.[3] Over the last few years as part of a DOD MANTECH, ATK Thiokol [4] and BAE Holston Army Ammunition Plant (HAAP) [5] have produced moderate quantities of TATB (~5 kg batches) with plans to scale up for DOD applications. Thiokol TATB is polycrystalline with an average particle size of about 40 μm (similar to WA TATB) but HAAP TATB is only 5-6 μm (similar to ultrafine). We have obtained small quantities of these materials for evaluation. [6]

Scope of work:

The project (1) compares new FK-800 with old Kel-F 800 and FK-800 lots currently available at LLNL, (2) compares and characterizes new TATB with old TATB, (3) formulates new FK-800 with wet-aminated TATB and new TATBs in according to HAAP slurry coating procedure into LX-17-2, and (4) evaluates the mechanical and detonation performance characteristics of this insensitive high explosive (IHE). Priorities are to prove that these new materials can be formulated, pressed to density and machined; and that they contain no impurities which might cause compatibility issues. Since 3M [1, 2], LANL [7], Pantex [8] and AWE [9, 10] are currently evaluating the new FK-800, we plan to share data rather than repeating their work. Our effort is described briefly below.

Task 1. Evaluation of newer characterization methods to identify structural variations between old and new Kel-F 800 including: Rheological and mechanical properties, copolymer content, degree of crystallinity, and interfacial interactions with TATB.

Task 2. Evaluate TATBs using scattering techniques to replace sieving operations called out in the specification [11] for particle size distribution measurements. Use SEM and OM for morphological differences between the old and new explosives. Evaluate the compaction characteristics of new TATB.

Task 3. Formulation of new LX-17-2 (with new FK-800 and/or new TATB)

Task 4. Evaluate mechanical and performance properties of LX-17-2. At a minimum, compressive strength, dynamic mechanical behavior and a 2" cylinder shot should be performed and compared with existing data for LX-17-1.

Results and Discussion:

Task 1. Characterization of Kel-F 800.

New and old lots of Kel-F 800 have been characterized for rheological and mechanical properties in the quarterly report, "Characterization and Qualification new TATB and Kel-F 800 for LX-17," dated February 16, 2007 [12].

Task 2. Characterization of new TATB.

The following four TATBs have been evaluated: New BAE Holston, new ATK Thiokol, wet-aminated (WA), and dry-aminated (DA) TATB. Small scale safety testing has been completed. Particle morphology was evaluated using Optical and SEM micrographs. Pressing characteristics of the dry powder as a function of pressure were performed. Ultra small angle x-ray scattering measurements on powder and pressed pellets were made. ATK TATB density distributions were compared to that of WA TATB using a gradient column approach. See "Characterization of new TATBs" [13] for results.

Task 3. Formulate LX-17-2 using FK-800 lot 1 with wet-aminated TATB and new TATBs.

A. Formulation and Processing

LX-17-2 was initially formulated according to the Pantex operating procedure for making LX-17-1 [14], scaling the quantities of materials back from 250 lbs to 50g using the Holston Reactor as shown in Figure 1.

The lacquer used for the slurry coating process was prepared by dissolving FK-800 in ethyl acetate (15% solids) overnight at room temperature using a magnetic stir bar. Once the lacquer was prepared, 200 ml of deionized water was charged into the Holston

Reactor vessel. Scaling the amount of water back from a 250 lb batch to a 50 g batch required only 50 ml of water per the Pantex operating procedure; however, 200 ml was necessary to bring the water level up to about 1/3 full, which is necessary for adequate mixing. TATB was then dispersed in the water by mixing as shown in Figure 2. Next, enough ethyl acetate was added (20 g) to the TATB slurry to saturate the mixing water, while continuing to stir. The FK-800/ethyl acetate lacquer was then added to the Holston Reactor vessel while stirring and excess ethyl acetate was used to ensure complete transfer of the lacquer. Heat was applied from a circulating bath through the heating jacket of the reactor vessel to bring the temperature up to 32-38°C. Water (50 g) was added to the slurry and vacuum was applied to evaporate off the ethyl acetate and to form the PBX granules. The solvent was further evaporated by raising the temperature of the slurry to 60°C. Once the granules hardened, the mixture was transferred into ice water as shown in Figure 3 and filtered through a Buchner funnel with vacuum to remove the residual water and solvent as shown in Figure 4. The granules were dried to constant weight overnight in an oven at 75-80°C.

The first four formulations (-ATK, -BAE, -WA1, and -WA2) shown in Table 1 were formulated using this procedure. All formulations use new FK-800 lot #1. The first LX-17-2 formulation is made up of new TATB from ATK Thiokol. The second LX-17-2 formulation is made up of new TATB from BAE Holston. The last three LX-17-2 formulations use wet-aminated TATB lot # C-061 (-WA1, -WA2, and -WA3).

Figure 5 shows the slurry coated granules from the first three formulations. These granules are commonly referred to as molding powder. LX-17-2 made with ATK Thiokol TATB yielded good granules, similar to typical LX-17-1 with minimal fines. LX-17-2 made with BAE Holston TATB contained a substantial amount of fines. LX-17-2 made with wet-aminated TATB also contained a substantial amount of fines. Differences in bead size are probably due to the initial particle size and morphology of the TATB crystals. The bulk density of each material is shown in Table 1. The specification for LX-17 High Explosive Molding Powder states that LX-17 shall be a minimum of 0.80 g/cc when tested for bulk density [15]. The values for bulk density for LX-17-2 formulations are lower than the specification for LX-17 probably because of the differences in processing.

Scaling the Pantex procedure down to 50 grams has its limitations. During the slurry coating process, build-up of TATB and binder on the walls of the reactor vessel was observed and product yields were as low as 75%. The large amount of build up on the reactor vessel walls may be due to strong adhesion of the binder with heated surfaces. To overcome this possible issue, the slurry coating process was performed without heating. The amount of ethyl acetate added to the TATB slurry was reduced from 20 g to 18 g and no extra ethyl acetate was used to wash down the sides during the process. Instead, a solution with the same ratio of ethyl acetate to water as already in the slurry was used to wash down the walls of the reactor during the process. The amount of ethyl acetate was reduced so that granules would form immediately after the FK-800/ethyl acetate lacquer was added to the TATB slurry. Instead of applying heat to drive off the solvent and harden the granules, excess water was added (150 g total) and vacuum was applied.

Sticking of the TATB and binder on the walls of the reactor was reduced substantially. However, there was still some sticking caused by splashing during mixing. This process was used for making the last batch of LX-17-2 with wet-aminated TATB (-WA3). As shown in Figure 6, the granule quality was improved resulting in less fines and higher yields. It would appear that using the new FK-800 lot 1 polymer has not adversely affected the PBXing process for LX-17.

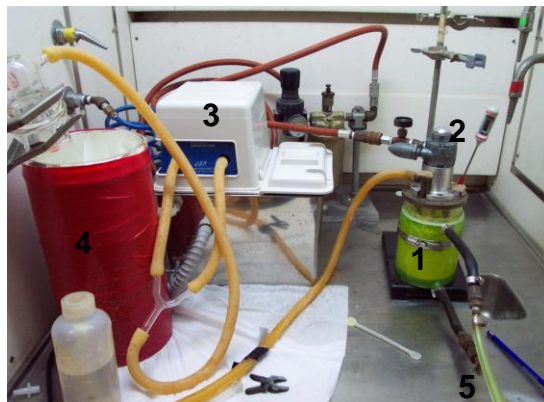


Figure 1. Slurry Coating Setup:
(1) Holston reactor, (2) Air motor, (3) Aspirator,
(4) Vacuum trap, (5) Heater hoses



Figure 2. TATB dispersed in water
Using Holston Reactor (LX-17-2-ATK)



Figure 3. LX-17-2 beads cooled with ice water

after solvent driven off (LX-17-2-ATK)



Figure 4. Granules filtered using Buchner funnel with vacuum (LX-17-2-ATK)

Table 1. LX-17-2 formulations (92.5% TATB/7.5% FK-800)

ID	Notebook No.	TATB Type	FK-800	Bulk Density (g/cc)	Comments
LX-17-2-ATK	SD-I-13	ATK Thiokol (C-562)	Lot 1	0.66	Pantex procedure used
LX-17-2-BAE	SD-I-16	BAE Holston (C-559)	Lot 1	0.48	Pantex procedure used
LX-17-2-WA1	SD-I-18	Wet-aminated (C-061)	Lot 1	0.51	Pantex procedure used
LX-17-2-WA2	SD-I-27	Wet-aminated (C-061)	Lot 1	0.63	Pantex procedure used
LX-17-2-WA3	SD-I-29	Wet-aminated (C-061)	Lot 1	0.64	No heat during slurry coating



Figure 5. a) LX-17-2-ATK (made with ATK Thiokol TATB); b) LX-17-2-BAE (made with BAE Holston TATB); c) LX-17-1-WA1 (made with Wet-aminated TATB lot C-061)

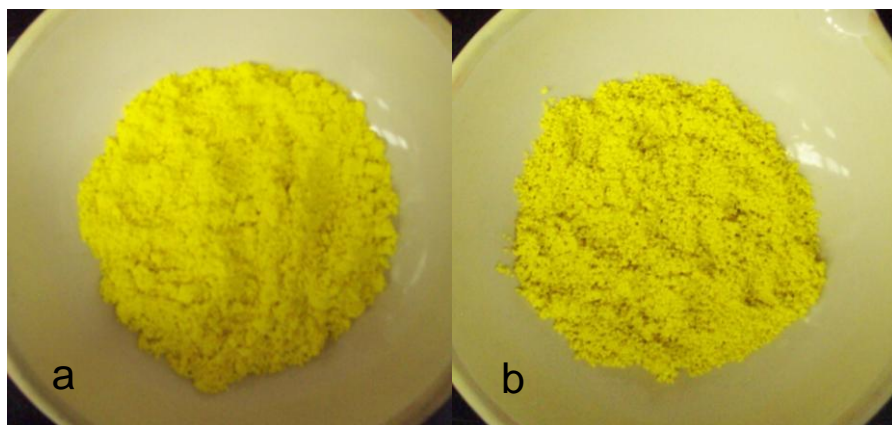


Figure 6. a) LX-17-2-WA2 (made using Pantex procedure); b) LX-17-2-WA3 (made using modified procedure without the use of heat)

B. Small-scale Safety Testing

It is important to characterize the safety and relative sensitivity of explosives. Small-scale safety testing including drop hammer tests, BAM friction tests, Electrostatic Spark tests, chemical reactivity tests, and Differential Scanning Calorimetry (DSC) are used to characterize the sensitivity of explosives here at LLNL. Below is a brief description of each test. Table 2 shows the results of the small-scale safety testing for each LX-17-2 formulation along with typical values for LX-17-1 [16,17]. All batches have similar impact, friction, and spark sensitivity, and chemical reactivity to LX-17-1. The DSC onset of decomposition temperature varies. The DSC onset temperature listed in Table 2 is defined here as the intersection between the baseline and the tangent line that can be drawn at the point of inflection on the edge of the decomposition peak. Typical LX-17-1 which includes wet-aminated TATB has an onset decomposition temperature of about 377°C [17] and pure wet-aminated TATB has a similar onset temperature. LX-17-2 made with both ATK Thiokol TATB and BAE Holston TATB have lower onset of decomposition temperatures, 362°C and 361°C respectively. These correspond to the onset temperatures of the pure TATBs which are 360°C for ATK Thiokol TATB and 359°C for BAE Holston TATB. The lower onset temperatures for the pure TATBs are probably due to their level of purity. See “Characterization of New TATBs” for further details [13]. LX-17-2 batches made with wet-aminated TATB and FK-800 lot 1 have similar onsets of decomposition to LX-17-1. See Figure 7 for an overlay of the DSC profiles for the five formulations. It appears that using FK-800 lot #1 as the binder in the LX-17-2 formulations does not affect the sensitivity of the explosives.

Drop Hammer Tests: Impact sensitivity is measured using the LLNL small-scale drop-hammer impact sensitivity test [18]. With this test, a 2.5 kg weight is dropped from different preset heights onto a 35 mg sample of explosive. Reaction or no reaction, which is based on the intensity of the sound emitted by the impact, is recorded. The impact sensitivity is summarized as DH50, the height in centimeters where the probability of explosion is 50%.

BAM Friction Tests: The Jules-Peters BAM machine [19] measures how an explosive reacts to shear stress. A 5 mg sample is scraped by a ceramic knob dragged sideways on a ceramic plate by a load weight ranging from 0.5 kg to 36 kg. The relative frictional sensitivity is based on the largest load where less than two reactions occur after ten trials.

Electrostatic Discharge Tests: The spark sensitivity test [20] is used to determine sensitivity to electrostatic discharge. With this test, a small sample of 3-5 mg is sealed in a plastic ring attached to a steel disk. A specified voltage is discharged through the sample. If no event is detected after ten trials, the explosive is designated as insensitive to spark. If an event is detected, the voltage level is lowered until 10 trials results in no events.

Chemical Reactivity Tests: The Chemical Reactivity Test (CRT) [21] is a method used for characterizing the thermal stability of explosives. Using a sealed stainless steel container in a silicone oil bath, a 0.25 g sample is heated to 120°C for 22 hours under 1 atm of He. Specific gases that evolve from the explosive during this heating process are analyzed including N₂, O₂, CO, NO, CO₂, and N₂O. Total gas evolution of more than 4 cc/g at 120°C is considered suspect and additional testing must be done to determine its thermal stability.

Differential Scanning Calorimetry (DSC): Differential Scanning Calorimetry (DSC) [22] is used to help determine the thermal properties of an explosive. DSC allows for the measurement of the difference in heat flow between the sample and an inert reference as a function of time and temperature. Observable thermal events that take place can be quantified and the onset of the decomposition exotherm can be used as an indication of thermal stability. During this test, approximately 0.5 mg of sample is heated at a linear heating rate of 10 °C/min using an aluminum pan with a pinhole in the cover.

Table 2. Small-scale safety data for LX-17-2 molding powder

ID	DH50 (cm)	Friction @ 36 kg	Spark (1 J)	CRT @ 120°C (g/cc)	DSC Onset Temp (°C)
Typical LX-17-1	>177.4	0/10	0/10	0.20	377
LX-17-2-ATK	>177.4	0/10	0/10	0.15	362
LX-17-2-BAE	>177.4	0/10	0/10	0.18	361
LX-17-2-WA1	>177.4	0/10	0/10	0.11	375
LX-17-2-WA2	>177.4	0/10	0/10	0.07	380
LX-17-2-WA3	>177.4	0/10	0/10	0.12	382

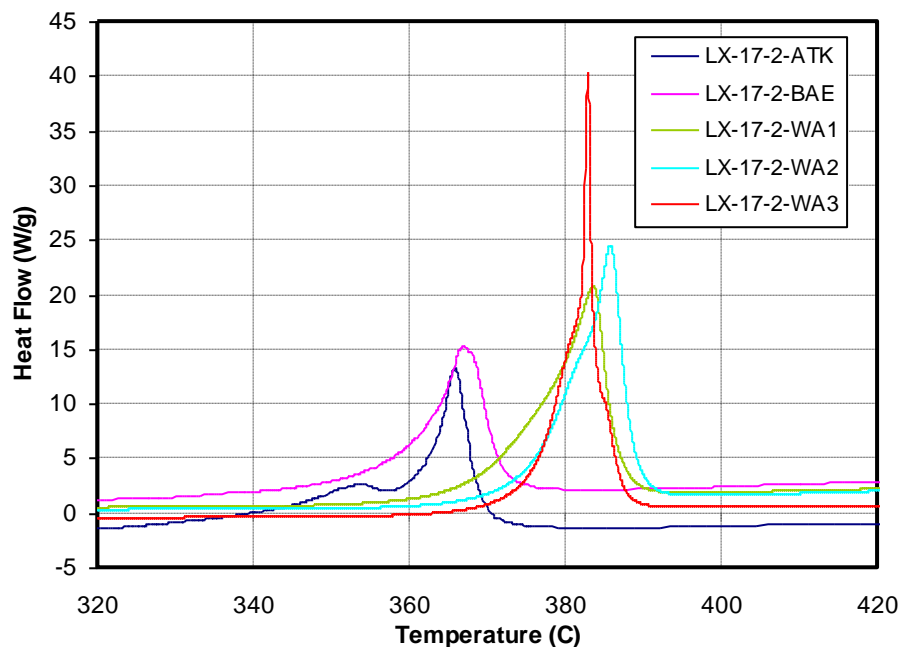


Figure 7. DSC profiles for LX-17-2 formulations

Task 4. Mechanical properties of LX-17-2

A. Pressing Characteristics

LX-17-2 formulations (-ATK, -BAE, -WA2) were compression molded into 0.5 in. diameter by 1 in. long right cylinders in a mechanical die. Six different sets of pressing conditions were used. These conditions along with the resulting pressing densities and their percentage of the Theoretical Maximum Density (% TMD) are shown in Table 3. The TMD for LX-17-1 is 1.943 g/cc. Typically, LX-17-1 is pressed at 30 kpsi, 105°C with 4 five minute cycles. Resulting pressing densities range from 97% to 98% TMD using a mechanical die (LX-17-1 Lot #C-329). LX-17-2 made with ATK Thiokol TATB gave pressing densities of 1.840 g/cc (94.7% TMD) or less. The lower than expected densities are probably due to the properties of the TATB. Based on the pressing study, optimal pressing conditions for maximum density for LX-17-2 made with Thiokol TATB are 30 kpsi, 100°C, with 3 three minute cycles. With LX-17-2 made with Thiokol TATB, higher densities were obtained at lower temperatures, which was unexpected. This temperature density relationship was not seen for any of the other formulations. Densities of 1.876 g/cc (96.6% TMD) or less were obtained for LX-17-2 made with the BAE Holston TATB; still lower than typical LX-17-1. Optimal pressing conditions for LX-17-2 made with BAE Holston TATB are 30 kpsi, 120°C, with 3 three minute cycles. Again, the lower densities are probably due to properties of the TATB. Pressing studies completed on neat TATB show higher densities for ATK Thiokol TATB, than for BAE Holston TATB [13]. Further studies would need to be done to address why neat TATB from ATK Thiokol can be pressed to higher density than TATB from BAE Holston, but when formulated into LX-17-2, the formulations containing BAE Holston TATB have

the higher densities. LX-17-2 made with wet-aminated TATB (-WA2) was also pressed at similar conditions to LX-17-2 made with Thiokol and Holston TATB. It can be used as a baseline for the kinds of densities that would be expected under those conditions. LX-17-2 made with wet-aminated TATB pressed to 1.889 g/cc (97.2% TMD) or less; the highest density under the conditions tested, which corresponds with pure wet-aminated TATB having higher pressing densities than the other TATBs. At optimal pressing conditions, LX-17-2 made with wet-aminated TATB has a density similar to LX-17-1, which shows that using FK-800 lot 1 does not affect its compaction density. However, the pressed parts did contain some small cracks with less pressing cycles, which also show up in their values for density. No pressing studies were completed on LX-17-1 using the same pressing conditions to determine if LX-17-1 would also crack with less pressing cycles.

LX-17-2 made with wet-aminated TATB, batch -WA1, was also compression molded. However, at the conditions listed in Table 3, the part would not hold together. It laminated into about 3-5 pieces. The part would only hold together after pressing it at typical LX-17-1 conditions of 30 kpsi, 105°C with 4 five minute cycles. Under these conditions, the resulting density, which was 97.3% TMD, was similar to typical LX-17-1. Due to a limited amount of material, no further studies could be done to determine why this batch of LX-17-2 did not press similarly to the other batches.

Table 3. Densities obtained during pressing study of LX-17-2 formulations

Pressing Conditions	-ATK	-BAE	-WA2
	Density (g/cc) / %TMD		
a. 20 kpsi, 100°C, 3x3 min dwell	1.829 / 94.1	1.853 / 95.4	1.882 / 96.9
b. 20 kpsi, 120°C, 3x3 min dwell	1.826 / 94.0	1.865 / 96.0	1.874 / 96.4
c. 30 kpsi, 100°C, 3x3 min dwell	1.840 / 94.7	1.866 / 96.1	1.887 / 97.1
d. 30 kpsi, 120°C, 3x3 min dwell	1.825 / 93.9	1.876 / 96.6	1.889 / 97.2
e. 30 kpsi, 100°C, 1x3 min dwell	1.823 / 93.8	1.852 / 95.3	1.870 / 96.2*
f. 30 kpsi, 120°C, 1x3 min dwell	1.813 / 93.3	1.863 / 95.9	1.860 / 95.7*

*small cracks visible after pressing

B. Compression Testing

Mechanical properties were measured in compression at a strain rate of 0.0001 sec⁻¹ using an MTS 858 Mini Bionix II test system that has a 5,500-lbs static force capacity (axial), and a 4 inch stroke. The environmental chamber used to control temperature was a MTS model 651.05 with a temperature range of -75°C to 275°C. Compression testing was done on 0.5 in. diameter by 1 in. long right cylinders made from the three different formulations of LX-17-2 that were pressed under the conditions listed in Table 3. Figures 8, 9, and 10 show the compression behavior at 23°C, 74°C, and -30°C respectively of LX-17-2 formulations compared to compression data for LX-17-1 taken previously [23]. LX-17-2 made with BAE Holston TATB has greater stiffness and substantially higher strength than LX-17-2 made with ATK Thiokol TATB, LX-17-2 made with wet-aminated TATB, and LX-17-1 at all temperatures tested. Both LX-17-2 made with Thiokol TATB and LX-17-2 made with Holston TATB have higher strength

and stiffness than LX-17-2 made with wet-aminated TATB. The differences in compression behavior between LX-17-1 and LX-17-2 made with wet-aminated TATB is probably due to the difference in degree of crystallization of the binder. Materials that undergo mechanical properties testing soon after being raised above their binder melting point from pressing will have a lower degree of crystallization of the binder causing lower strength and stiffness during mechanical properties testing.

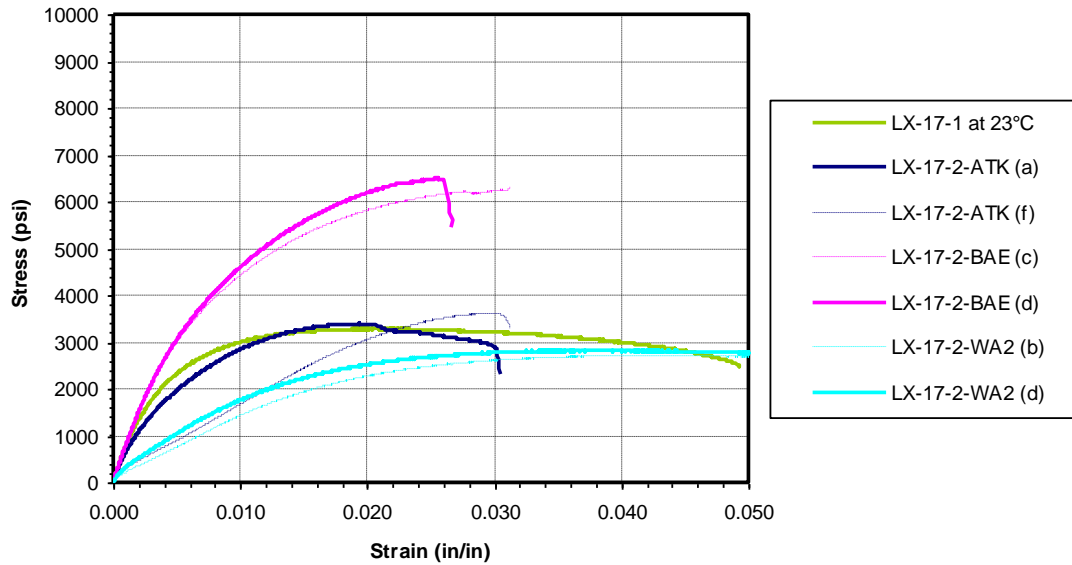


Figure 8. Compression tests completed on the different LX-17-2 formulations and LX-17-1 at a strain rate of 0.0001/s at 23°C

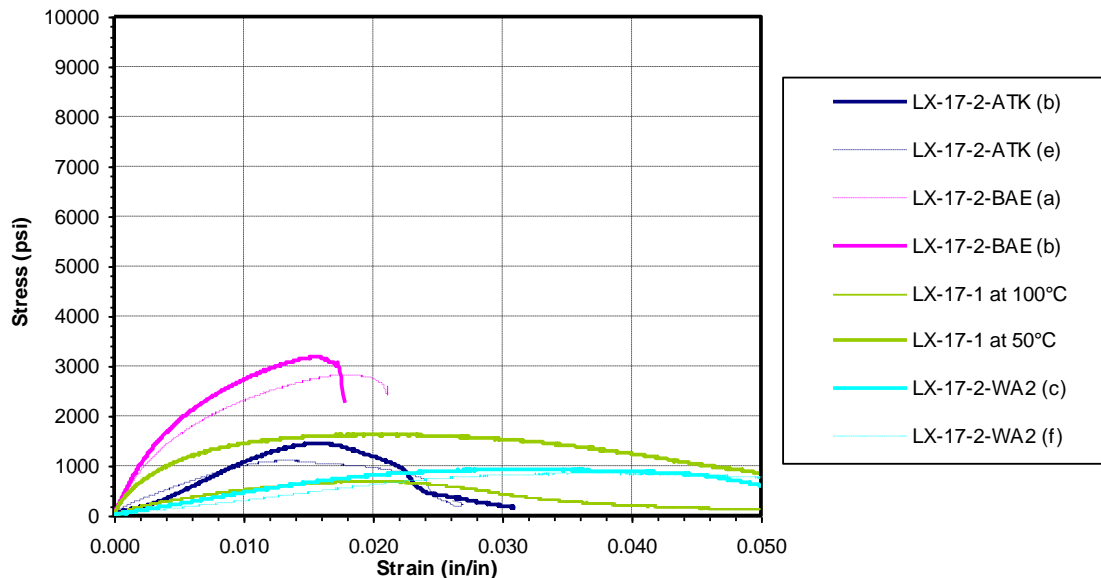


Figure 9. Compression tests completed on the different LX-17-2 formulations at 74°C compared to LX-17-1 at 50°C and 100°C (strain rate = 0.0001/s)

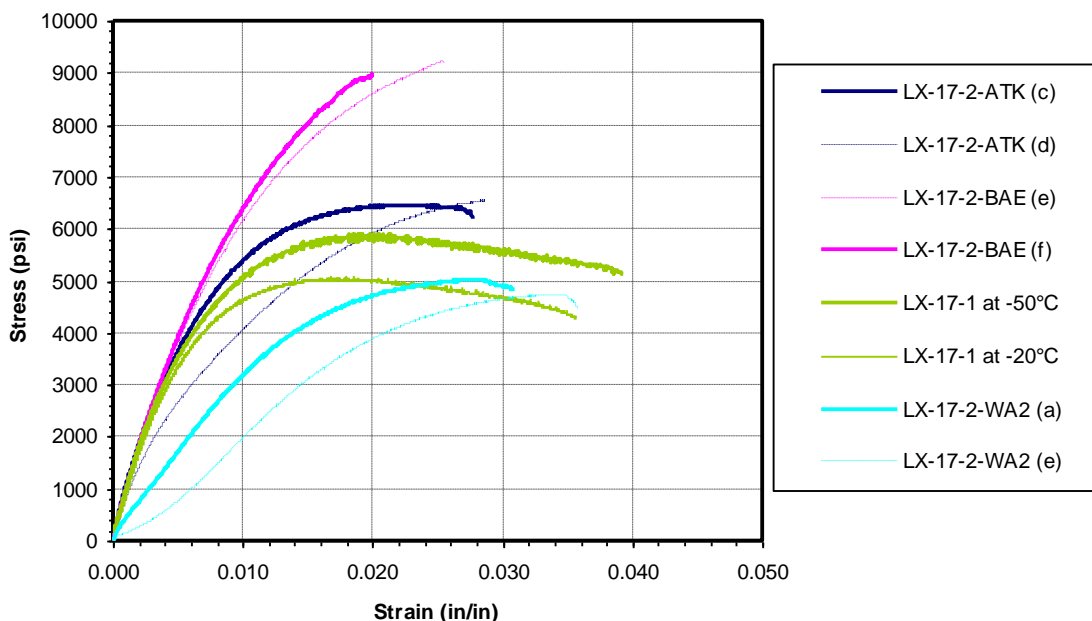


Figure 10. Compression tests completed on the different LX-17-2 formulations at -30°C compared to LX-17-1 at -50°C and -20°C (strain rate = 0.0001/s)

C. Thermal Expansion and Ratchet Growth

Attempts to measure the thermal expansion of LX-17 and PBX-9502 over multiple cycles of temperature have found irreversible volume expansion [24-28]. The term “ratchet growth” can be used to describe the behavior of compacted TATB based explosives when they are temperature cycled because they tend to irreversibly grow with each cycle. TATB based explosives expand in response to heating and contract on cooling. However, they do not contract back to their initial dimensions. They retain some of the growth over each cycle. Figure 11 shows the ratchet growth behavior typical for LX-17-1.

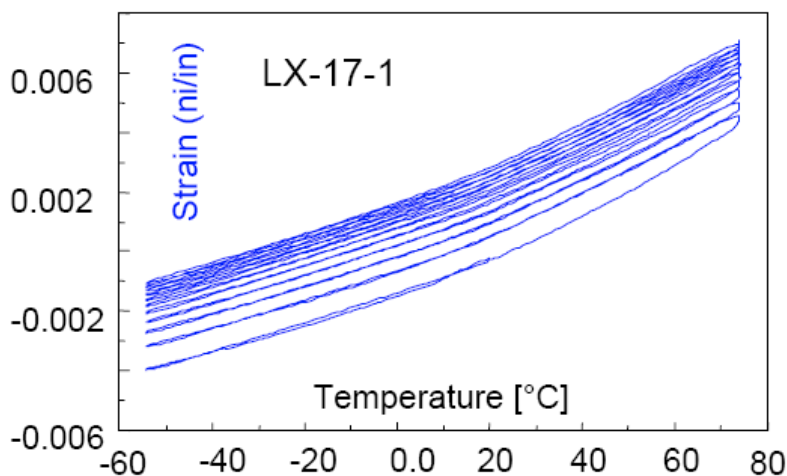


Figure 11. Typical ratchet growth behavior for LX-17-1

Using the optimal pressing conditions for each formulation, LX-17-2 -ATK, -BAE, and -WA1 were compression molded into 1/4 in. diameter by 1/4 in. long right cylinders in a mechanical die with densities of 1.81g/cc, 1.88g/cc, and 1.87g/cc respectively. These compacted samples were evaluated for ratchet growth using a thermal-mechanical analyzer (TMA), model 2940 manufactured by TA instruments, which continuously monitors a samples axial dimension via a quartz probe linked to an LVT. The materials were cycled from -54°C to 74°C at 3°C per minute and the temperature was held for 10 minutes at each end of the cycle. Figures 12, 13, and 14 show the thermal expansion behavior of each material. Unlike LX-17-1, the LX-17-2 formulations do not undergo irreversible growth. In fact, the LX-17-2 made with ATK Thiokol TATB shows some shrinkage over time. This may be due to the lower compaction densities.

It is also typical of LX-17-1 to continue to expand during the 10 minute isotherms at 74°C. During the 10 minute isotherms at -54°C, it is also typical to see minimal expansion and no contraction. The LX-17-2 materials differ from LX-17-1 in that they not only expand during the 10 minute isotherms at 74°C, but they also continue to contract at the 10 minute isotherms at -54°C.

To confirm the unusual results, LX-17-2 made with wet aminated TATB (-WA1) that was compression molded into a 0.5 in. diameter by 1 in. long right cylinder in a mechanical die with a density of 1.89g/cc was temperature cycled from -54°C to 74°C in a Thermal Product Solutions Tenney Chamber. Before and after 30 cycles, the dimensions of the right cylinder were measured. The dimensions in the axial direction decreased by 0.03%, supporting that no axial ratchet growth is observed. However, the radial dimensions increased by 0.29%, which suggests that there is some ratchet growth in the radial direction. Past attempts at measuring thermal expansion of LX-17-1 have shown greater axial expansion than radial expansion due to a small degree of alignment of the TATB molecules under uniaxial compression [26]. Further studies would need to be done to determine why the typical ratchet growth was not observed in these new formulations.

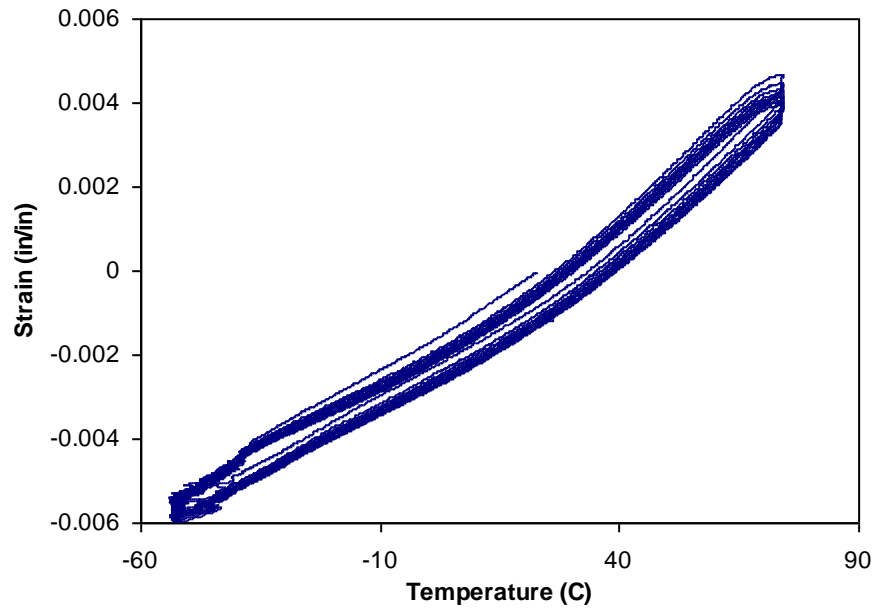


Figure 12. Thermal Expansion behavior of LX-17-2 made with Thiokol TATB (-ATK) shows some shrinkage instead of ratchet growth

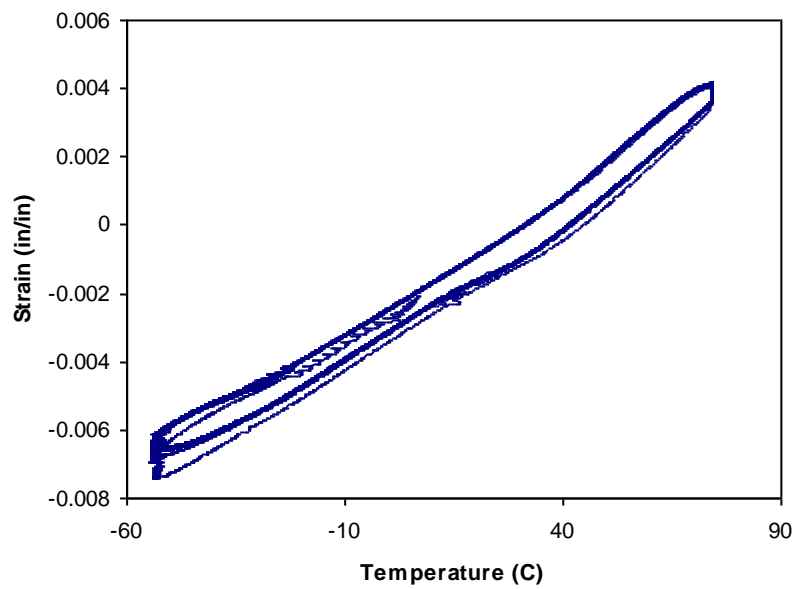


Figure 13. Thermal Expansion behavior of LX-17-2 made with Holston TATB (-BAE) shows little to no ratchet growth

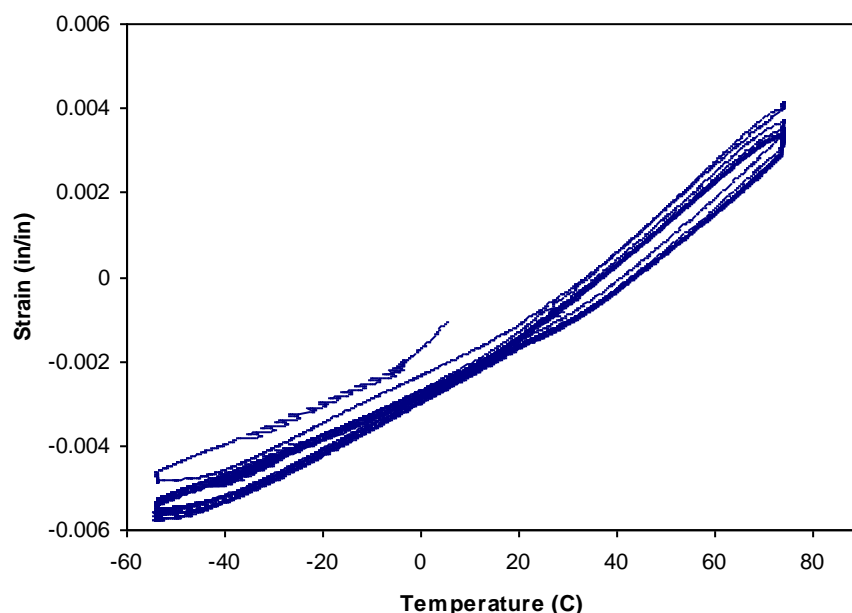


Figure 14. Thermal Expansion behavior of LX-17-2 made with Wet-aminated TATB (-WA1) does not undergo ratchet growth

Conclusions:

LX-17-2 formulations containing FK-800 lot 1 and new TATBs have been made on a 50 gram scale successfully using the Holston slurry coating process. Safety testing shows equivalent sensitivity to LX-17-1 for friction, spark, and impact. LX-17-2 formulations containing the new TATBs have a lower onset of decomposition as evaluated using DSC, which corresponds to the onsets of decomposition for the pure TATBs. Pressing studies show that LX-17-2 made with wet-aminated TATB and FK-800 lot 1 have substantially equivalent compaction properties to LX-17-1. LX-17-2 made with the new TATBs can not be pressed to as high as density as LX-17-1, probably because of the properties of the neat TATB. Compression tests show that LX-17-2 made with BAE Holston TATB has higher strength and stiffness than LX-17-2 made with ATK Thiokol TATB, LX-17-2 made with wet-aminated TATB, and LX-17-1. LX-17-2 formulations do not show ratchet growth typical of LX-17-1. Further studies are recommended to address unusual results for ratchet growth.

Acknowledgements:

Special thanks to Franco Gagliardi and Bruce Cunningham, for pressing and compression testing and insights into mechanical properties, to Sally Weber for pressing, to Gary Hust who ran small scale-safety testing and helped with slurry coating, to Rosalind Swansiger and Pat Lewis for help with slurry coating, to Heidi Turner who ran DSC and TMA, and to Cathy Bartholdi who ran chemical reactivity tests.

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